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IS 5960 (Part 4) : 1997

ISO 1444: 1996

भारतीय मानक

माँस और माँस के उत्पाद — परीक्षण पद्धतियाँ

भाग 4 मुक्त वसा अंश ज्ञात करना

(पहला पुनरीक्षण)

Indian Standard MEAT AND MEAT PRODUCTS — METHODS OF TEST

PART 4 DETERMINATION OF FREE FAT CONTENT

(First Revision)

(ISO TITLE : MEAT AND MEAT PRODUCTS — DETERMINATION OF FREE FAT CONTENT)

ICS 67.120.10

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MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

NATIONAL FOREWORD

This Indian Standard (Part 4) which is identical with ISO 1444: 1996 'Meat and meat products — Determination of free fat content', issued by the International Organisation for Standardization (ISO), was adopted by the Bureau of Indian Standards on the recommendation of the Slaughter House and Meat Industry Sectional Committee and approval of the Food and Agriculture Division Council.

The text of ISO standard has been approved as suitable for publication as Indian Standard without deviations. Certain conventions are however not identical to those used in Indian Standards. Attention is particularly drawn to the following:

- a) Wherever the words 'International Standard' appear referring to this standard, they should be read as 'Indian Standard'; and
- b) Comma (,) has been used as a decimal marker while in Indian Standards, the current practice is to use a point (.) as the decimal marker.

This Indian Standard was originally published in 1970 based on the method adopted by the ISO/TC 34/SC 6. On a review by the Technical Committee responsible for formulating standards in the area of meat and meat products, a need was felt to revise the standard aligning it with ISO 1444: 1996. This would ensure uniformity in test methodology and find wider acceptance.

In reporting the result of a test or analysis made in accordance with the standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2: 1960 'Rules for rounding off numerical values (revised)'.

CROSS REFERENCES

International Standard	Indian Standard	Degree of Correspondence
ISO 3100-1:1991 Meat and meat products — Sampling and preparation of test samples — Part 1: Sampling	IS/ISO 3100-1:1991 Meat and meat products — Methods of sampling	Identical
ISO 1442: 1996 Meat and meat products — Determination of moisture content (Reference method)	IS 5960 (Part 5): 1971* Methods of test for meat and meat products: Part 5 Determination of moisture content	Technically Equivalent
ISO 5725-1: 1994 Accuracy (trueness and precision) of measurement methods results — Part 1: General principles and definitions	IS 5420 (Part 1): 1969 Guide on precision of test method: Part 1 Principles and applications	Technically Equivalent

The International Standards referred in Annex A are for information and guidance.

^{*} Under revision for alignment with ISO 1442: 1996.

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Indian Standard MEAT AND MEAT PRODUCTS — METHODS OF TEST

PART 4 DETERMINATION OF FREE FAT CONTENT

(First Revision)

1 Scope

This International Standard specifies a method for the determination of the free fat content of meat and meat products by means of extraction.

2 Normative reference

The following standard contains provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the edition indicated was valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent edition of the standard indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 1442:1996¹⁾, Meat and meat products — Determination of moisture content (Reference method).

3 Definitions

For the purposes of this International Standard, the following definitions apply.

- **3.1** free fat content of meat and meat products: Mass of the fat extracted under the conditions specified in this International Standard divided by the mass of the test portion. The free fat content is expressed as a percentage by mass.
- **3.2 test result:** The value of a characteristic obtained by carrying out a specified test method.

[ISO 5725-1]

Extraction, by means of *n*-hexane or light petroleum, of the dried residue obtained in accordance with the method of determination of the moisture content specified in ISO 1442. Removal of the solvent by evaporation, then drying and weighing of the extract.

5 Reagent and material

5.1 Extraction solvent, *n*-hexane or, alternatively, light petroleum distilling between 40 °C and 60 °C, and having a bromine value less than 1. For either solvent, the residue on complete evaporation shall not exceed 0,002 g per 100 ml.

5.2 Boiling-chips

6 Apparatus

Usual laboratory apparatus and, in particular, the following:

- **6.1** Homogenizing equipment, mechanical or electrical, capable of homogenizing the test sample. This includes a high-speed rotational cutter, or a mincer fitted with a plate with holes not exceeding 4,5 mm in diameter.
- **6.2 Extraction thimble**, made of filter paper and defatted.
- 6.3 Cotton wool, defatted.

⁴ Principle

¹⁾ To be published. (Revision of ISO 1442:1973)

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6.4 Extraction apparatus, continuous or semicontinuous, for example the Soxhlet type.

NOTE 1 Instead of the classical Soxhlet technique, the extraction procedure may also be performed with extraction systems capable of simultaneous extraction of a number of samples, such as Soxtec or other similar automated instruments.

- **6.5** Sand bath or water bath, electrically heated, or similar suitable apparatus.
- **6.6 Drying oven**, electrically heated, capable of being maintained at 103 °C ± 2 °C.
- **6.7 Desiccator**, containing an efficient desiccant, e.g. silica gel.
- **6.8** Analytical balance, capable of weighing to an accuracy of ± 0.001 g.

7 Sampling

It is important that the laboratory receive a sample which is truly representative and has not been damaged or changed during transport or storage.

Sampling is not part of the method specified in this International Standard. A recommended sampling method is given in ISO 3100-1.

The mass of the laboratory sample shall be not less than $200\,\mathrm{g}$.

Store the sample in such a way that deterioration and change in its composition are prevented.

8 Preparation of test sample

- **8.1** Homogenize the test sample with the appropriate equipment (6.1). Take care that the temperature of the sample material does not rise above 25 °C. If a mincer is used, pass the sample at least twice through the equipment.
- **8.2** Fill a suitable airtight container with the prepared sample. Close the container and store in such way that deterioration and change in composition are prevented. Analyse the sample as soon as practicable, but always within 24 h of homogenization.

9 Procedure

NOTE 2 If it is required to check whether the repeatability requirement is met, carry out two single determinations in accordance with 9.1 and 9.2 under repeatability conditions.

9.1 Test portion

Take a known mass of 5 g to 8 g, weighed to the nearest 0,001 g (m_0) of the prepared sample and dry it by the procedure specified in ISO 1442. If desired, the dried test portion from the determination of moisture content may be used for the determination of free fat.

For reliable measurements, the lowest level of fat present in the test portion should be 0,05 g.

9.2 Determination

Dry the flask of the extraction apparatus (6.4), containing some boiling-chips (5.2), for 1 h in the drying oven (6.6) set at 103 °C. Allow the flask to cool to room temperature in the desiccator (6.7) and weigh to the nearest 0,001 g (m_1) .

Transfer the dried test portion (9.1) quantitatively from the dish to the extraction thimble (6.2). Remove the last traces of the dried test portion from the dish, using cotton wool (6.3) moistened with the extraction solvent (5.1), and also transfer this cotton wool to the thimble. Place the thimble in the extraction tube of the apparatus. Pour the extraction solvent into the flask of the extraction apparatus; the amount of solvent shall be at least one and a half to two times the capacity of the extraction tube of the apparatus. Fit the flask to the extraction apparatus. Heat the flask for at least 6 h on the sand bath or water bath (6.5), according to the extraction rate and the apparatus used.

When a Soxtec or other similar automatic procedure is used, the heating period shall be at least 2 h.

After extraction, take the flask containing the liquid from the extraction apparatus and distil off the solvent using, for example, the sand bath or water bath. Evaporate the last traces of solvent using air blowing if desired.

Dry the flask for 1 h in the oven (6.6) set at $103 \,^{\circ}$ C and, after allowing it to cool to room temperature in the desiccator (6.7), weigh to the nearest 0,001 g. Repeat the operations of heating, cooling and weighing until the results of two successive weighings, separated by 1 h of heating, do not differ by more than 0,1 % of the mass of the test portion (m_2) .

Verify completion of the extraction by taking a second extraction flask and extracting for a further period of 1 h with a fresh portion of the solvent. The increase in mass shall not exceed 0.1 % of the test portion.

10 Calculation

Calculate the free fat content, w_f , as a percentage by mass, using the following equation:

$$w_{\rm f} = \frac{(m_2 - m_1)}{m_0} \times 100 \%$$

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where

- m_0 is the mass, in grams, of the test portion taken for drying;
- m_1 is the mass, in grams, of the extraction flask with boiling chips;
- m₂ is the mass, in grams, of the flask and boiling chips with the fat, after drying.

Report the result rounded to one decimal place.

11 Precision

The precision of the method has been established by an interlaboratory test (see reference [4]), carried out in accordance with ISO 5725 $^{2)}$. For the values obtained for the repeatability limit, r, and the reproducibility limit, R, a probability level of 95 % holds.

11.1 Repeatability

The absolute difference between two independent single test results, obtained using the same method on identical test material in the same laboratory by the same operator using the same equipment within a short interval of time, should not be greater than the repeatability limit r as calculated using the following equation:

$$r = -0.05 + 0.06 \,\overline{w}_{\rm f}$$

where $\overline{w}_{\rm f}$ is the mean of the two test results, expressed as a percentage by mass.

11.2 Reproducibility

The absolute difference between two single test results, obtained using the same method on identical test material in different laboratories with different operators using different equipment, should not be greater than the reproducibility limit R as calculated using the following equation:

$$R = 0.04 + 0.06 \,\overline{w}_t$$

where $\overline{w}_{\rm f}$ is the mean of the two test results, expressed as a percentage by mass.

12 Test report

The test report shall specify:

- the method in accordance with which sampling was carried out, if known;
- the method used;
- the test result(s) obtained; and
- if the repeatability has been checked, the final quoted result obtained.

It shall also mention all operating details not specified in this International Standard, or regarded as optional, together with details of any incidents which may have influenced the test result(s).

The test report shall include all information necessary for the complete identification of the sample.

²⁾ ISO 5725:1986 was used to obtain the precision data. This has now been cancelled and replaced by ISO 5725-1:1994 and subsequent parts.

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Annex A

(informative)

Bibliography

- [1] ISO 3100-1:1991, Meat and meat products Sampling and preparation of test samples Part 1: Sampling.
- [2] ISO 5725:1986, Precision of test methods Determination of repeatability and reproducibility for a standard test method by inter-laboratory tests.
- [3] ISO 5725-1:1994, Accuracy (trueness and precision) of measurement methods and results Part 1: General principles and definitions.
- [4] FOSTER, M.L. and SHARON, E.G. Soxtec fat analyzer for determination of total fat in meat: Collaborative Study. J. Assoc. Off. Anal. Chem., 75, 1992, pp. 288-292.

Bureau of Indian Standards

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Review of Indian Standards

Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the latest issue of 'BIS Handbook' and 'Standards: Monthly Additions'.

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Amendments Issued Since Publication

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Northern : SC	O 335-336, Sector 34-A, CHANDIGARH 160022	$\left\{\begin{array}{c} 60\ 38\ 43 \\ 60\ 20\ 25 \end{array}\right.$
Southern: C.	I. T. Campus, IV Cross Road, CHENNAI 600113	{ 235 02 16, 235 04 42 235 15 19, 235 23 15
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